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(72) Inventor: YANG, Huimin; 7240 North Pierron Road, Glendale, WI 53209 (US).

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(74) Agents: WAGNER, Alan, E. et al.; Whyte Hirschboeck Dudek, S.C., 111 E. Wisconsin Avenue, Suite 2100, Milwaukee, WI 53202 (US).

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(71) Applicant: BRADY WORLDWIDE, INC. [US/US];  
2221 West Camden Road, P.O. Box 571, Milwaukee, WI  
53201-0571 (US).

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(54) Title: LOW OUTGASSING PRESSURE SENSITIVE ADHESIVES AND METHOD OF USE

(57) Abstract: An improved pressure sensitive adhesive (PSA), comprising at least one polymer selected from natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes or combinations thereof; and at least one tackifying resin, was disclosed which uses a scavenger to capture impurities inherent in the polymer and/or the tackifying resin. Additionally, a method for making improved, cleaner computer devices by using the improved PSA on computer device components that contain a PSA. The component thus produced was ideally suited to be used in disk drives. Also disclosed were computer devices, preferably computer data storage devices, made by the method disclosed.

LOW OUTGASSING PRESSURE SENSITIVE ADHESIVES AND METHOD OF USE

This invention relates to pressure sensitive adhesives (PSA). More particularly, this invention relates to PSA's for use with computer devices, and to methods for preparing cleaner computer devices, such as cleaner data storage devices.

Computer disk drives were complex assemblages of surface treated alloys, plastics, elastomeric, and ceramic parts containing various lubricants and a variety of adhesives. Pressure sensitive adhesives were used for various applications such as seals, labels, damping, etc. in today's computer data storage devices. The use of PSA's has been an effective means of lowering unit cost for storage devices. A typical drive may include at least one component which bears a PSA, e.g., a tape, which was used to hold the housing of the disk drive together.

Demand for increased storage capacity of disk drives has produced product designs requiring a higher level of concern for adhesive contamination. New disk drive technology, such as decreasing flying heights, use of magneto-resistive head technology, and pseudo-contact recording, have improved the performance and capacity of disk drive components. Use of this new technology has also left the disk drive more susceptible to damage from environmental factors. Building drives with contaminated or outgas-prone parts can result in stiction/wear and functional problems, including electrical error issues from thermal asperities.

The cleanliness of PSA materials has been a concern for disk drive engineers. Physical contamination such as dust particles, skin flake, and moisture, along with possible chemical contamination by the materials used in the PSA, can affect the drive

life or reliability of the device. Adhesive materials can deposit contaminants on disk and reading heads and cause reading problems or disk crashes.

Specific ions and organic tins were particularly harmful to heads and disks. Small levels of chlorine containing materials have been identified to be responsible for disk 5 corrosion. Organic materials capable of undergoing polymerization were unacceptable at very low levels. Organic or inorganic acids or bases can corrode the sensitive layers of the storage disk. Typical types of microcontamination commonly found in the disk drive industry include organic contamination that can cause stiction; corrosion from residual anions (particularly, chloride and sulfate ions); outgassing, which can result in stiction; 10 and airborne particulates.

Polyacrylate-based PSA's have been used extensively in the components for disk drives. A select few were considered useable; none were totally satisfactory. The chemical byproducts from the initiators used in the initial polymerization of acrylates can be unacceptable even at relatively low levels in disk drives. The solvents and their 15 impurities used to dissolve and coat polyacrylate PSA's can be a problem if not thoroughly dried from the finished adhesive coating. Most polyacrylate pressure sensitive adhesives must be chemically cross-linked to provide the necessary performance characteristics. The chemicals used for, or byproducts produced from, the cross-linking can be unacceptable or at unacceptable levels. The levels of unreactive monomers and 20 their impurities in polyacrylate PSA's have been known to cause drive failures.

The disk drive industry was resorting to two strategies in order to minimize the presence of microcontaminants in the environment of the disk drive. The first strategy was to use one of a variety of cleaning methods in order to remove the microcontaminants from the finished disk drive parts. Typically, such cleaning methods were either aqueous

cleaning, solvent cleaning, or carbon dioxide cleaning. All of these cleaning methods have their advantages and difficulties. However, no cleaning method can remove all contaminants. Cleaning was a percentage removal process, and it targets specific contaminants. The higher the initial level of contaminants, the higher the final level of contaminants in the finished product.

5           The second strategy used by the disk drive industry was to use parts and processes that contain or produce fewer contaminants. These more stringent specification requirements were a challenge to the suppliers of components for the disk drive industry. Specific requirements include low outgassing and low ionic contamination. For example, 10 a typical limit for outgassed materials may be as low as 2500 nanograms per square centimeter ( $\text{ng}/\text{cm}^2$ ); the limit for anions may be a maximum of 800  $\text{ng}/\text{cm}^2$ .

Further disclosure on microcontaminants in the disk drive industry can be found in Peter Mee et al, Management of Disk Drive Component Microcontamination, IDEMA® Insite, Vol. IX, No. 2 (March/April 1997).

15           It has been a difficult challenge for the adhesive industry to meet the requirements of the disk drive engineers with polyacrylate adhesives. This invention relates to a method for preparing a computer device in a manner that a lower level of microcontaminants was present around the disk drive.

In one embodiment, the invention comprises an improved PSA comprising:

- 20           A. at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof;
- B. at least one tackifying resin; and
- C. at least one scavenger.

In another embodiment, the invention comprises an improved method for preparing a computer device which includes at least one component bearing a PSA, the method comprising the steps of:

- A. forming a solution by dissolving in a solvent
  - (1) at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof,
  - (2) at least one tackifying resin, and
  - (3) at least one scavenger, wherein at least one of the polymer or the tackifying resin includes at least one impurity, the impurity comprising a compound with at least one polar reactive moiety;
- B. contacting the scavenger with the impurity to form a reaction product;
- C. applying the filtered solution to a substrate;
- D. drying the solution on the substrate; and
- E. applying the substrate to a computer device.

In yet another embodiment, the invention comprises a computer device comprising at least one component containing a PSA, wherein the PSA comprises:

- A. at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof;
- B. at least one tackifying resin; and
- C. at least one scavenger.

As used herein, the term "styrenic" refers to mono-alkenyl arenes. Thus, a styrenic polymer or copolymer was formed by the polymerization of at least one mono-alkenyl arene.

A "block copolymer" was a polymer containing long stretches of two or more monomeric units linked together by chemical valences in one single chain, such that the long monomeric stretches alternate with each other in which a block(s) of one type of polymer structure was attached to a block(s) of another type of polymer structure. A "diblock copolymer" was a block copolymer that has the general structure A-B, where A was a long stretch of one homopolymer structure and B was a long stretch of a second homopolymer structure. A "triblock copolymer" was a block copolymer that has the general structure A-B-A, where A was a long stretch of one homopolymer structure and B was a long stretch of a second homopolymer structure.

A "tackifying resin" was a resin that, when added to a rubber or an elastomer, the resulting composition has the properties of a pressure sensitive adhesive. "Pressure sensitive adhesives" were permanently and aggressively tacky (sticky) solids which form immediate bonds when two parts were brought together under pressure. For pressure sensitive adhesives, "tack" can be described as the property whereby the adhesive will adhere tenaciously to any surface with which it comes into contact under light pressure. The strength of the bond will be greater under increasing pressure, hence the term pressure sensitive. Tack can be quantified as the force required to separate an adherend and an adhesive at the interface shortly after they have been brought rapidly into contact under a light load of short duration. Tack can be measured by using the ASTM D-2979 procedure.

A "scavenger" was a substance added to a system or mixture to consume or inactivate traces of impurities.

A "hydrocarbon resin" was a resin in the number molecular weight range of a few hundred up to about 8,000, which was obtained or synthesized from rather basic hydrocarbonaceous materials such as petroleum, coal, tar, and turpentine.

An improved computer device was prepared by preparing at least one adhesive as described below and affixing the adhesive to at least one component of the computer device. Preferably, the component was a film, tape or label which was used to seal or identify one or more components of the computer device. The computer device was preferably a disk drive or other data storage device.

#### The Pressure Sensitive Adhesive

The adhesive used in this invention comprises (i) at least 10, preferably at least 40, and not in excess of 90, preferably not in excess of 75, weight percent of a polymer, (ii) at least about 10, preferably at least 25, and not in excess of 90, preferably not in excess of 60, weight percent of a tackifying resin, and, (iii) at least 0.1, preferably at least 2, more preferably at least 3, and not in excess of 8, preferably not in excess of 6, more preferably not in excess of 4 weight percent of a scavenger, all percentages based on the total weight of the adhesive.

#### Polymers

The PSA of this invention comprises polymers typically known in the art of making PSAs. In general, such polymers impart an elastic or viscoelastic property to the PSA. Polymers that were useful for the PSAs of this invention include rubbers, such as natural rubber, and elastomers, such as styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof. Certain block copolymers have

been found useful for making PSAs and, preferably, the polymer of this invention was at least one hydrogenated block copolymer.

Block copolymers were well-known in the field of PSAs. Any one of these well-known block copolymers can be included within the composition of this invention, including those described in some detail within U.S.P. 3,239,478 and 3,917,607. These particular block copolymers typically take on the general configuration A-B-A or A-B-A-B, wherein each "A" block, which was generally characterized as an end block, was a thermoplastic polymer block prepared by a polymerization of a styrenic monomer such as styrene,  $\alpha$ -methyl-styrene, tert-butyl-styrene, and vinyl-toluene.

The elastomer "B" blocks, which were characteristically identified as "mid blocks," were prepared by the propagation of a polymer chain of a conjugated diene such as butadiene or isoprene from the end of the end block already synthesized. Then either using sequential monomer addition or a coupling agent, the desired block copolymers, A-B-A or A-B-A-B, were generated. If the block copolymers, A-B-A or A-B-A-B were hydrogenated, a saturated mid block would be obtained. Such block copolymers, which were prepared by known procedures, include styrene-butadiene-styrene block copolymer (SBS), styrene-isoprene-styrene block copolymer (SIS), styrene-ethylene butylene-styrene block copolymer (SEBS), styrene-ethylene propylene-styrene (SEPS), and  $\alpha$ -methylstyrene-ethylene propylene- $\alpha$ -methylstyrene. A typical molecular weight for such block copolymers was between 10,000 and 500,000, expressed as number average molecular weight.

Preferably, the block copolymer of this invention was a hydrogenated block copolymer. Hydrogenation minimizes the presence of double bonds in potentially outgassed materials that could result in those outgassed materials polymerizing on the

computer device components. Such polymerization can interfere with the recording, storage or reading of data in a computer data storage device.

The hydrogenated block copolymer was preferably a block copolymer of polystyrene and a polydiene, the polydiene typically selected from the group consisting of 5 polybutadiene and polyisoprene, wherein the unsaturated mid block of either polybutadiene or polyisoprene was hydrogenated to yield a saturated mid block. The saturated mid block of the hydrogenated block copolymer has the structure of poly(ethylene-propylene), poly(ethylene-butylene), or both.

More preferably, the block copolymers of this invention comprise styrene end 0 blocks and hydrogenated polybutadiene and/or hydrogenated polyisoprene mid blocks. Such block copolymers were also disclosed in U.S.P. 4,136,699; 4,361,672; 4,460,364; 4,714,749; and 5,459,193, and in KRATON® Thermoplastic Rubbers published by Shell Chemical Company.

Most preferably, the block copolymer has end blocks of styrene whose number 5 average molecular weight was in the range of 10,000 to 30,000, and the mid block was a block of hydrogenated polyisoprene having a number average molecular weight of 125,000. Such hydrogenated block copolymers were known as styrene-ethylene-propylene-styrene (SEPS) copolymers. Such block copolymers were commercially available from the Shell Chemical Company under the trademark KRATON® G.

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The KRATON® G hydrogenated block copolymers have a number average molecular weight ( $M_n$ ) of 25,000 to 300,000, as measured by gel permeation chromatography (GPC). Among the KRATON® G polymers, the most preferred were KRATON® G1650, KRATON® G1652, KRATON® G1654, KRATON® G1657, and

KRATON® G1730, or a combination thereof. More information on KRATON® G polymers was disclosed in KRATON® Polymers for Adhesives and Sealants, available from Shell Chemical Company.

In one preferred embodiment of the invention, the block copolymer, in combination with the tackifying resin(s), outgasses less than 1500 ng/cm<sup>2</sup> as measured by 5 Modified IDEMA MII-98 (described later). Optionally, the outgassing content of the polymer can be reduced by applying removal means to the polymer prior to the scavenging reaction. Such removal means can comprise extreme high temperature drying, spray drying, aqueous cleaning, solvent cleaning, or CO<sub>2</sub> cleaning.

10 A further embodiment of this invention comprises using a polyurethane as the polymer. Polyurethanes were thermoplastic polymers produced by the condensation reaction of a polyisocyanate and a hydroxyl-containing material, e.g., a linear polyester or polyesters containing hydroxyl groups. The basic polymer unit was formed as shown in Formula I:



15 where R<sub>1</sub> and R<sub>2</sub> were hydrocarbon radicals or substituted hydrocarbon radicals. Such polymers were discussed in U.S.P. 4,661,542. An example of such a polyurethane was 20 ESTANE® 5703 which was commercially available from BF Goodrich.

Natural rubber was a well-known material. Natural rubber was a polyisoprene derived from latex obtained from Hevea trees, coagulated with acetic or formic acid. Natural rubber can also be made synthetically.

Acrylate copolymers were known in the art. Such acrylic adhesives were discussed in U.S.P. 5,981,666.

#### Tackifying Resins

Tackifying resins of this invention were resins that associate predominantly with the elastomeric block or mid block and were substantially incompatible with nonelastomeric or end blocks. These mid block associating resins were compatible with the mid block in that between 100 and 200 or more parts by weight of the mid block associating resin show a clear film when the particular mid block associating resin was combined with 100 parts of the mid block of the block copolymer and cast from solution in toluene.

In one embodiment of this invention, the tackifying resins usable in this invention, in combination with the block copolymer(s), outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98 (described later). Optionally, the outgassing content of the tackifying resins can be reduced by applying removal means to the tackifying resins prior to the scavenging reaction. Such removal means can comprise extreme high temperature drying, spray drying, aqueous cleaning, solvent cleaning, or CO<sub>2</sub> cleaning.

Examples of tackifying resins that may be useful in this invention include polyhydric esters of rosin or hydrogenated rosin esters, such as glycerol and pentaerythritol esters of hydrogenated rosins and of highly stabilized rosins, esters of polyhydric alcohol, synthetic polyterpenes, terpene-olefin copolymers, terpene-phenols, tall oil rosin, synthetic saturated hydrocarbon resins, such as saturated alicyclic hydrocarbons, olefinic resins, aromatic containing resins, phenol-aldehyde resins,  $\alpha$ -pinene resins,  $\beta$ -pinene resins, terpene-phenolic resins, and copolymers such as of 1,3-pentadiene and 2-methyl-2-butene, or mixtures thereof. Other examples of tackifying

resins were disclosed in U.S.P. 4,361,663 and 4,399,249, and in U.S.P. 4,136,699; 4,361,672; 4,714,749; and 5,459,193.

The preferred tackifying resin of this invention was of the type known as "hydrocarbon resins". A good description of hydrocarbon resins can be found in Kirk-Othmer, Encyclopedia of Chemical Technology, Second Edition, Vol. 11, Interscience, New York, 1966, pg. 242. Many of the so-called hydrocarbon resins commercially available today were terpene resins, i.e., polymers with (repeating) isoprene ( $C_5H_8$ ) or  $C_{10}H_{16}$  units. These polymers can be natural or synthetic and can be copolymers (including terpolymers, etc.), since isoprene was an olefin which can be copolymerized with other olefins. Terpene-phenols were also produced.

Aromatic monomers useful in forming the aromatic containing resin compositions of this invention can be prepared from any monomer containing substantial aromatic qualities and a polymerizable unsaturated group. Typical examples of such aromatic monomers include: styrenic monomers, e.g., styrene,  $\alpha$ -methylstyrene, vinyl toluene, methoxy styrene, tertiary butyl styrene, chlorostyrene, etc.; indene monomers including indene, methyl indene and others. Aliphatic monomers were typically natural and synthetic terpenes which contain  $C_6$  and  $C_5$  cyclohexyl or cyclopentyl saturated groups that can additionally contain a variety of substantial aromatic ring substituents.

Aliphatic tackifying resins can be made by polymerizing a feed stream containing sufficient aliphatic monomers such that the resulting resin exhibits aliphatic characteristics. Such feed streams can contain other aliphatic unsaturated monomers such as 1,3-butadiene, cis-1,3-pentadiene, trans-1,3-pentadiene, 2-methyl-1,3-butadiene, 2-methyl-2-butene, cyclopentadiene, dicyclopentadiene, terpene monomer, terpene phenolic resins and others. Mixed aliphatic aromatic resins contain sufficient aromatic monomers

and sufficient aliphatic monomers to produce a resin having both aliphatic and aromatic character. The article by Davis, "The Chemistry of C<sub>5</sub> Resins," discusses synthetic C<sub>5</sub> resin technology. The preferred tackifying agents were hydrogenated C<sub>5</sub> to C<sub>12</sub> resins, preferably a C<sub>9</sub> resin.

5 Representative examples of useful aliphatic resins include hydrogenated synthetic C<sub>9</sub> resins, synthetic branched and unbranched C<sub>5</sub> resins and mixtures thereof. Representative examples of aromatic tackifying resins include styrenated terpene resins, styrenated C<sub>5</sub> resins or mixtures thereof.

10 Preferably, the tackifying resin was derived by the polymerization and hydrogenation of pure monomer hydrocarbon feed stocks (wherein the hydrocarbon monomer has 5 or 9 carbon atoms). Such hydrocarbon resins were highly stable, light colored, low molecular weight, non-polar resins and were suggested for use in plastics, adhesives, coatings, sealants and caulk. Hydrogenation minimizes the presence of double or triple bonds in potentially outgassed materials that could result in those 15 outgassed materials polymerizing on the computer device components. Such polymerization can interfere with the recording, storage or reading of data in a computer data storage device.

20 More preferably, the tackifying resin of this invention was low molecular weight nonpolar hydrocarbon resin commercially available under the trademark REGALREZ® from Hercules Incorporated. Most preferably, the tackifying resin was at least one of REGALREZ® 1018, REGALREZ® 1085, REGALREZ® 1094 and REGALREZ® 1126.

Scavengers

In an electronic assembly, pure hydrocarbons may be tolerated to a relatively higher level than that of the chemicals containing reactive moieties, such as acid, alcohol or amine, which were undesirable, if not outright unacceptable, even at relatively low levels. Some of these chemicals containing reactive moieties can be substantially removed from film-type polymers or formulated adhesives by appropriate drying conditions. However, chemicals containing reactive moieties may not be fully removed during drying, especially as the thickness of the adhesive was increased. These chemicals may then slowly escape from the adhesive to become microcontaminants in a computer device.

This invention reduces the level of these chemicals that was outgassed, from a polymer or an adhesive composition, through an "anchoring reaction". The anchoring reaction was a reaction between reactive groups on the scavengers and reactive moieties on the outgassing chemicals. The products of these anchoring reactions were either less susceptible to outgassing, or were less reactive if they do outgas, or both. In this invention, the anchoring reaction was typically conducted in a solution prepared by dissolving or suspending the PSA components in a suitable liquid, such as, e.g., toluene. While the anchoring reaction was normally substantially completed in the solution phase, it was also possible for trace levels of scavengers to continue reacting at the elevated temperatures that occur during drying.

The scavengers of this invention react with impurities, such as acids, amines and/or alcohol, at moderate temperatures, preferably room temperature, and for short time periods. The products resulting from this anchoring reaction must be non-volatile even at

elevated temperatures such as 85 °C. The scavengers must be compatible with the PSA system.

Typical scavengers of this invention were low molecular weight compounds and oligomers that contain groups that react with the reactive moieties of the impurities or have the ability to absorb chemical impurities. The scavengers used in this invention were typically chosen based on the impurities present in the adhesive composition or constituents of the adhesive. Mixtures of scavengers can provide for the capture of multiple impurities.

Preferably, the scavengers of this invention have moieties that react with the polar reactive moieties (such as acid, alcohol, amine, etc.) present in typical outgassed materials found in PSAs. Examples of scavengers usable in this invention include polyisocyanates, aziridines, aluminum oxide ( $\text{Al}_2\text{O}_3$ ), metallic hydrides, monohydrogen silicides, and mixtures thereof. Preferred scavengers were polyisocyanates and aluminum oxide.

Suitable polyisocyanates include the DESMONDUR™ series polymers commercially available from Mobay Corporation, preferably, DESMONDUR™ 3300 and DESMONDUR™ 3390. DESMONDUR™ 3300 and DESMONDUR™ 3390 have the preferred characteristics of relatively high isocyanate content and low free monomers content.

The PSA of this invention was conveniently prepared by adding at least 0.1, preferably at least 2, more preferably at least 3, up to 8, preferably not to exceed 6, more preferably not to exceed 4 weight percent, based on the weight of the total PSA, of the scavenger(s) to an adhesive system, stirring for a period of time, filtering or decanting the adhesive when necessary, coating the PSA solution onto a substrate, then drying properly. Typically, a filtering or decanting step was only needed to remove the anchoring reaction products formed when aluminum oxide or similar absorbent was used as a scavenger.

Optional Components

The PSA's of this invention can optionally include other components known in the art. These components can include plasticizing oils, resin modifiers, and antioxidants.

A "plasticizing oil", also known as an extending oil, was an organic compound added to a high polymer both to facilitate processing and to increase the flexibility and toughness of the final product by internal modification (solvation) of the polymer molecule. The latter was held together by secondary valence bonds; the plasticizing oil replaces some of these with plasticizing oil-to-polymer bonds, thus aiding movement of the polymer chain segments. Among the more important plasticizing oils were nonvolatile organic liquids and low-melting solids, e.g., phthalate, adipate, and sebacate esters, polyols, such as ethylene glycol and its derivatives, tricresyl phosphate, castor oil, etc.

These optional components tend to be "dirty" in that they contain undesirable ions and/or contribute to outgassing from the PSA. Antioxidants in particular can be harmful if outgassed into a disk drive system. Therefore, the current preferred embodiments of the invention do not include these optional components. However, the invention contemplates use of these ingredients at levels wherein the PSA containing these ingredients outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98 (described later).

Method of Forming the PSA

The PSA compositions of hydrogenated block copolymers and tackifying resins can be formed by technologies well known in the art, such as the technologies disclosed in U.S.P. 4,361,672. Examples of suitable methods for forming PSA's include, *inter alia*:

5 (i) compounding on a hot two-roll mill; (ii) melting the block copolymer and the tackifying resin and mixing the melted components until homogeneous; (iii) other methods employed in the plastic and elastomer industries, such as high shear intensive mixing, twin screw extrusion or tandem extrusion techniques; and (iv) dissolving the mixtures in suitable organic solvents such as toluene and heptane, taking care to form

10 homogeneous solutions that were then coated on a substrate (e.g., on a film backing) before the solvent was evaporated.

Hot-melt compounding (any of methods i-iii, above) was not a favored embodiment of this invention. The heat from these methods can degrade the components of the PSA, thereby creating more material that can potentially outgas from the PSA.

15 However, the invention contemplates use of these methods provided that the resulting PSA outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98.

Preferably, the PSA of this invention was formed by dissolving the mixtures in suitable organic solvents taking care to form homogeneous solutions that were then coated on the component for the computer device before the solvent was evaporated.

20 Preferably, these solutions contain at least 10, more preferably at least 20, most preferably at least 30, up to preferably 80, more preferably not in excess of 60, most preferably not in excess of 50, weight percent solids based on the total weight of the solution.

Suitable organic solvents must be inert to the block copolymer and the tackifying resin. Additionally, the organic solvent must be selected such that substantially all of the

solvent can be evaporated from the PSA. "Substantially all of the solvent" refers to removal of the solvent such that the residual levels of solvent were less than 5 percent, preferably less than 2 percent of the total outgassed materials per Modified IDEMA M11-98. Preferred organic solvents include toluene, cyclohexane and heptane, more preferably toluene.

5            Optionally, the solution can be filtered or decanted after the scavenging reaction has been accomplished. This option was preferred when aluminum oxide or similar absorbent was used as a scavenger.

0            The solvent can be evaporated from the PSA by any technique known in the art, such as vacuum drying or, preferably, exposure to hot air in a drying oven. The temperature of the hot air must be maintained below the autoignition point of the solvent. The appropriate drying conditions were dependent on the substrate to which the PSA was applied and the process time available for drying. For example, when using a substrate comprising a Type S polyester film (available from Du Pont), the preferred hot air 5 temperature for evaporating toluene from the PSA of this invention was 250 °F to 350 °F for a drying time of 2 to 3 minutes. The practical upper limit on drying time was determined from the available equipment and the desired production rate.

In one preferred embodiment the PSA was formed by:

- A. forming a solution by dissolving in a solvent
- 0            (1) at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof, preferably the polymer was at least one of a hydrogenated styrene-elastomer-styrene block copolymer or a hydrogenated styrene-elastomer-styrene-elastomer block copolymer,

- (2) at least one tackifying resin, and  
(3) at least one scavenger, wherein at least one of the polymer or the tackifying resin includes at least one impurity, the impurity comprising a compound with at least one reactive moiety;
- 5      B. contacting the scavenger with the impurity to form a reaction product;  
C. removing the reaction product from the solution, when necessary;  
D. applying the solution to a substrate;  
E. drying the solution on the substrate; and  
F. applying the substrate to a computer device.

10       In another preferred embodiment, the substrate of the steps D and E, above, was a component of a computer device, more preferably, a component of a computer data storage device.

A hallmark of the PSA of this invention was that the PSA with a scavenger has lower outgassing, especially of chemicals harmful to a disk drive, than the same adhesive without a scavenger produced under the same drying temperature and time.

15       The following specific examples will more precisely describe the invention and teach the procedures presently preferred in practicing the same, as well as the improvements and advantages realized thereby. These examples were provided for illustration purposes only and shall not be construed to limit the scope of the subject  
20 matter of the invention.

### EXAMPLES

#### Example 1 - Measurement of Outgassed Materials

Outgassed materials were measured using the IDEMA M11-98 Dynamic Headspace Outgas Procedure with the following method details or exceptions (Modified IDEMA M11-98).

The permanent and expendable equipment (Section 2) consists of a: (a) Type 303 stainless steel cylindrical chamber with approximate internal dimensions: 1.25 inches deep by 2.25 inches in diameter; (b) Supelco<sup>1</sup> stainless steel thermal desorption tubes packed with the following sorbent: bed A= 100 milligrams Tenax TA, bed B= 250 milligrams Carbotrap B; (c) Hewlett-Packard<sup>2</sup> 6890 Gas Chromatograph/ 5973 Mass Spectrometer; (d) Perkin Elmer<sup>3</sup> ATD-400 Automated Thermal Desorption Unit; (e) Reztek<sup>4</sup> NTI<sup>®</sup>-5 gas chromatograph column, Reztek pn# 12223; and, n-hexadecane (Fisher Scientific<sup>5</sup> pn# 03035) in dichloromethane employed as external standard for semi-quantitation.

The sample collection and analysis (Section 3) was conducted as follows. The thermal desorption tubes were conditioned at 320 °C for 8 minutes. Desorption tubes were installed and the samples were placed in the sample outgassing chambers. The sample chamber flow rate was set at approximately 50 milliliters per minute (ml/min.) using 99.99 percent nitrogen gas. The outgassing chamber was continuously heated at 85 °C. The desorber was programmed to desorb at 320 °C for 8 minutes at 50 ml/min. The cold trap was set at -30 °C and desorbed at 350 °C, with a hold time of 8 minutes, and valve and line temperatures set at 200°C. The outlet split was set at 54 ml/min. The sample split ratio was approximately 49:1. The gas chromatograph flow rate was approximately 1 ml/min.

Semi-quantitation was accomplished by injecting 5 microliters of a 200 nanogram per microliter dichloromethane solution of n-hexadecane into a heated sample chamber

via an in-line injection port and outgassing onto reconditioned thermal desorption tubes for 185 minutes at 85 °C. The response factor was calculated by averaging the peak area of the total ion chromatogram for at least 6 replicates.

The amount of target compound or compounds was calculated (Section 4) by dividing the total ion count for the peak of the target compound or compounds by the external standard response factor and multiplying by 1,000 nanograms. The total outgassing for the sample was determined by integrating the top  $20 \pm 2$  peaks. The final result was expressed as nanograms per square centimeter where the sample area was determined by summing the area of both sides of a film sample.

#### Notes

- (1) Supelco, Supelco Park, Bellefonte, PA 16823-0048 USA.
- (2) Hewlett-Packard Company, Chemical Analysis Group, 2850 Centerville Road, Wilmington, DE 19808-1610 USA.
- (3) The Perkin-Elmer Corporation, 761 Main Avenue, Norwalk, CT 06859-0010 USA.
- (4) Reztek Corporation, 110 Benner Circle, Bellfonte, PA 16823 USA.
- (5) Fisher Scientific, 711 Forbes Avenue, Pittsburgh, PA 15219-4785 USA

#### Example 2 - Block Copolymer PSA Examples

A Comparative Sample (A) and three examples of the PSA of the invention (B, C, D) were prepared by dissolving the components in toluene to form a 35.5 percent solids solution. The formulas for each of these examples were shown in TABLE 1. None of the adhesive solutions of B, C or D were filtered or decanted.

TABLE 1 - Block Copolymer PSA Formulas

<u>Ingredient</u>	Purity	Chemistry/Function	A (g)	B (g)	C (g)	D (g)
KRATON® G1650	>99%	SEBS* polymer	1.7	1.7	1.7	1.7
KRATON® G1657	>99%	SEBS* polymer (29% diblock)	2.50	2.50	2.50	2.50
KRATON® G1730	>99%	SEPSEP** polymer	25.0	25.0	25.0	25.0
REGALREZ® 1085	NA	hydrocarbon tackifying resin	19.4	19.4	19.4	19.4
REGALREZ® 1018	NA	hydrocarbon tackifying resin	3.90	3.90	3.90	3.90
MORESTER® 49007 <sup>3</sup>	NA	optional modifier	2.63	2.63	2.63	2.63
DESMONDUR™ N3390	90% solution	polyisocyanate scavenger	0	1.70 <sup>1</sup>	0	0
DESMONDUR™ N3300	100%	polyisocyanate scavenger	0	0	2.20 <sup>2</sup>	0
Aluminum Oxide	>99%	scavenger	0	0	0	2.20 <sup>2</sup>

\* SEBS - polystyrene-(polyethylene-butylene)-polystyrene

\*\* SEPSEP - polystyrene-(polyethylene-propylene)-polystyrene-(polyethylene-propylene)

<sup>1</sup> 3 weight percent based on total composition weight.

<sup>2</sup> 4 weight percent based on total composition weight.

<sup>3</sup> MORESTER 49007 is a registered trademark owned by Morton International which was added as a modifier but has no effect on the final properties of the adhesive.

Each adhesive solution was coated onto a separate polyester film and dried at 300

°F for 3 minutes. The dried adhesive films were 1 mil thick. Then the coated polyester

film was die-cut into the same size specimens and their outgassing was tested using a gas

chromatograph-mass spectrometer according to Modified IDEMA M11-98. The results

of the outgassing measurements were shown in TABLE 2. These results demonstrate that

the use of a scavenger substantially decreases, by at least 16 percent, the amount of

outgassed material in a PSA comprising hydrogenated block copolymers.

TABLE 2 - Outgassing Results for Block Copolymer PSAs

Example	Scavenger	Scavenger (weight %)	Outgas (ng/cm <sup>2</sup> )	Outgas Reduction (%)
A	none	0	491	NA
B	DESMONDUR™ N3390	3	151	69
C	DESMONDUR™ N3300	4	412	16
D	aluminum oxide	4	358	27

Example 3 - Polyurethane PSA Examples

This example demonstrates the reduction of outgassed material in a polyurethane which can then be used to prepare a PSA. A high level of butylated hydroxytoluene (BHT) was measurable in ESTANE® 5703 (a polyurethane available from BF Goodrich) as shown in TABLE 3. A comparative example (E) was prepared by dissolving ESTANE® 5703 in dioxolane to form a 10 percent solution. An example of the invention (F) was prepared by making a solution containing 10 percent ESTANE® 5703, 0.5 percent of Al<sub>2</sub>O<sub>3</sub> and 89.5 percent of dioxolane, all percentages were by weight based on the total weight of the solution. Both Comparative Example E and Example F were stirred for 24 hours at room temperature. The Al<sub>2</sub>O<sub>3</sub> and the product of the scavenging reactions formed a precipitate from which the polymer solution was decanted. These solutions were carefully coated onto separate pieces of TESLINT™ paper (a porous polypropylene film available from DuPont). Preferably, the solution was uniformly absorbed into the paper. The coated paper samples were then dried at room temperature until no weight change was detected after 1 hour. The outgassing was measured according to Modified IDEMA M11-98 and the results were shown in TABLE 3. These

results demonstrate that the use of a scavenger of this invention also decreases outgassing in polyurethanes by at least 30 percent.

5 **TABLE 3 - Outgassing Results for Polyurethane**

	Example	Outgas Material (ppb)	Outgas Reduction (wt%)	BHT (ppb)	BHT Reduction (wt%)
ESTANE® 5703	E	$5.4 \times 10^6$	NA	$9.4 \times 10^5$	NA
ESTANE® 5703 with Al <sub>2</sub> O <sub>3</sub>	F	$3.4 \times 10^6$	37	$6.3 \times 10^5$	33

10 ESTANE® is a trademark of BF Goodrich.

## WHAT IS CLAIMED IS:

1. An improved pressure sensitive adhesive (PSA) comprising:
  - A. at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof;
  - B. at least one tackifying resin; and
  - C. at least one scavenger.
2. The PSA of Claim 1 wherein the polymer comprises at least one of a hydrogenated styrene-elastomer-styrene block copolymer or a hydrogenated styrene-elastomer-styrene-elastomer block copolymer.
3. The PSA of Claim 1 wherein the scavenger was selected from the group consisting of polyisocyanates, aziridines, aluminum oxide, metallic hydrides, monohydrogen silicides, and mixtures thereof.
4. The PSA of Claim 1 in which the PSA outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98.
5. The PSA of Claim 1 in which the PSA outgasses less than 500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98.
6. The PSA of Claim 1 in which the PSA outgasses at least 16 percent less material, as measured by Modified IDEMA M11-98, than a similar PSA, without a scavenger.
7. The PSA of Claim 2 in which the PSA outgasses at least 16 percent less material, as measured by Modified IDEMA M11-98, than a similar PSA, without a scavenger.

8. The PSA of Claim 2 in which the elastomer component of the hydrogenated styrene-elastomer-styrene block copolymer or the hydrogenated styrene-elastomer-styrene-elastomer block copolymer was composed of poly(ethylene-propylene) or poly(ethylene-butene) or both.
- 5 9. The PSA of Claim 2 in which the elastomer component of the hydrogenated styrene-elastomer-styrene block copolymer or the hydrogenated styrene-elastomer-styrene-elastomer block copolymer was made by the polymerization of one or more of (i) isoprene, or (ii) 1,3-butadiene.
- 10 10. The PSA of Claim 2 in which the PSA comprises between 10 to 90 weight percent of the block copolymer, between 10 to 90 weight percent of the tackifying resin, and between 0.1 to 10 weight percent of the scavenger, based on the weight of the PSA.
11. The PSA of Claim 1 in which the tackifying resin was a hydrocarbon resin.
12. The PSA of Claim 11 in which the tackifying resin was hydrogenated.
- 15 13. An improved method for preparing a computer device which includes at least one component bearing a PSA, the method comprising the steps of:
  - A. forming a solution by dissolving in a solvent
    - (1) at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof,
    - (2) at least one tackifying resin, and
    - (3) at least one scavenger, wherein at least one of the polymer or the tackifying resin includes at least one impurity, the impurity comprising a compound with at least one reactive moiety;

- B. contacting the scavenger with the impurity to form a reaction product;
  - C. applying the solution to a substrate;
  - D. drying the solution on the substrate; and
  - F. applying the substrate to a computer device.
- 5        14. The method of Claim 13 wherein the polymer was a hydrogenated styrene-elastomer-styrene block copolymer or a hydrogenated styrene-elastomer-styrene-elastomer block copolymer.
- 10      15. The PSA of Claim 13 wherein the scavenger was selected from the group consisting of polyisocyanates, aziridines, aluminum oxide, metallic hydrides, monohydrogen silicides, and mixtures thereof.
- 15      16. The method of Claim 13 further comprising the step of separating the reaction product from the solution prior to applying the solution to the substrate.
17. The method of Claim 13 in which the computer device was a data storage device.
18. The method of Claim 13 wherein the PSA outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98.
19. The method of Claim 13 in which the PSA outgasses at least 16 percent less material, as measured by Modified IDEMA M11-98, than a second PSA of the same composition except that the second PSA does not comprise a scavenger.
- 20      20. A computer device comprising at least one component containing a PSA, wherein the PSA comprises:
  - A. at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof;

- B. at least one tackifying resin; and
  - C. at least one scavenger.
21. The computer device of Claim 20 wherein the polymer comprises at least one of a hydrogenated styrene-elastomer-styrene block copolymer or a hydrogenated styrene-elastomer-styrene-elastomer block copolymer.
- 5
22. The computer device of Claim 20 in which the computer device was a computer data storage device.
23. The computer device of Claim 20 wherein the scavenger was selected from the group consisting of polyisocyanates, aziridines, aluminum oxide ( $\text{Al}_2\text{O}_3$ ), metallic hydrides, monohydrogen silicides, and mixtures thereof.
- 10
24. The computer device of Claim 20 wherein the PSA outgasses less than 1500 ng/cm<sup>2</sup> as measured by Modified IDEMA M11-98.
25. The computer device of Claim 20 in which the PSA outgasses at least 16 percent less material, as measured by Modified IDEMA M11-98, than a second PSA of the same composition except that the second PSA does not comprise a scavenger.
- 15
26. The computer device of Claim 21 in which the PSA outgasses at least 16 percent less material, as measured by Modified IDEMA M11-98, than a second PSA of the same composition except that the second PSA does not comprise a scavenger.
27. An improved PSA consisting essentially of:
- 20
- A. at least one polymer selected from the group consisting of natural rubber, styrenic-elastomer copolymers, acrylate copolymers, polyurethanes and combinations thereof;
  - B. at least one tackifying resin; and
  - C. at least one scavenger.

28. The PSA of Claim 27 wherein the polymer comprises at least one of a hydrogenated styrene-elastomer-styrene block copolymer or a hydrogenated styrene-elastomer-styrene-elastomer block copolymer.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/27260

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C09J153/02 C09J175/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C09J C09D C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, COMPENDEX, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 997 709 A (HUDDLESTON ELWYN ET AL) 5 March 1991 (1991-03-05) example 1 claims ---	1-12
X	US 4 080 348 A (KORPMAN RALF) 21 March 1978 (1978-03-21) column 2, line 43 - line 47 ---	1-12
X	WO 95 33794 A (KORPMAN RALF ASS) 14 December 1995 (1995-12-14) page 12, line 10 - line 15 claims 12,15 ---	1-12
X	US 5 807 637 A (JAUCHEN PETER ET AL) 15 September 1998 (1998-09-15) claims ---	1,3 -/-

 Further documents are listed in the continuation of box C. Patent family members are listed in annex.

## \* Special categories of cited documents :

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- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*&\* document member of the same patent family

Date of the actual completion of the international search

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Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Pollio, M

## INTERNATIONAL SEARCH REPORT

International Application No  
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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Section Ch, Week 199412 Derwent Publications Ltd., London, GB; Class A14, AN 1994-098036 XP002157346 & JP 06 049425 A (MINNESOTA MINING & MFG CO), 22 February 1994 (1994-02-22) abstract -----	1-28

## INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 00/27260

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 4997709	A	05-03-1991	NONE		
US 4080348	A	21-03-1978	AU	512345 B	09-10-1980
			AU	2478377 A	09-11-1978
			CA	1087341 A	07-10-1980
			DK	215677 A	19-11-1977
			FR	2352045 A	16-12-1977
			GB	1558220 A	19-12-1979
			IN	146069 A	17-02-1979
			JP	1554888 C	23-04-1990
			JP	52140544 A	24-11-1977
			JP	61006872 B	01-03-1986
WO 9533794	A	14-12-1995	US	5760135 A	02-06-1998
			AU	2660695 A	04-01-1996
US 5807637	A	15-09-1998	DE	19614620 A	23-10-1997
			DE	59701798 D	06-07-2000
			EP	0801121 A	15-10-1997
			ES	2147957 T	01-10-2000
			JP	10036780 A	10-02-1998
JP 6049425	A	22-02-1994	NONE		